Analytical Accuracy of Directly Fused Rock Specimen by Electron Probe Microanalyser

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By

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Abstract

Analytical technique of whole rock analysis using electron probe on the glass prepared by a direct fusion method, and the result of analyses are reported. Based on the analyses of the Geological Survey of Japan standard rocks, JB-1 and JG-1, accidental and systematic errors are evaluated. For most of the major elements our data show good agreement with the recommended values. And the release of alkalis by vaporization during heating is concluded to be negligible.

I. Introduction

Methods of major element analysis of bulk rock using electron probe micro-analyser (EPMA) have been proposed by several investigators (GULSON and LOVERING, 1968; RUCKLIDGE *et al.*, 1970; MORI *et al.*, 1971; NICHOLLS, 1974; FUKUYAMA and SAKUYAMA, 1976). We are currently using the direct fusion of powdered rock as described by NICHOLLS (1974). The following describes the accuracy and precision of analyses performed in our laboratory.

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II. Sample preparation and analytical method

Rock samples were crushed to pass 200–300 mesh. About 30 mg of samples were fused on an iridium strip to yield glasses, at 1600°C to 1800°C. Melts were stirred on the iridium strip for about 30 seconds with a platinum rod. Under the microscope, unmelted primary crystals or quench crystals were not observed. For glasses of basalt (JB–1) and granite (JG–1), scanning by EPMA did not detect any inhomogeneity (Fig. 1).

The glasses were analysed by EPMA model JXA–50A with 35° take-off angle. Instrumental conditions were as follows; 15 KV accelerating voltage, 0.02 μA

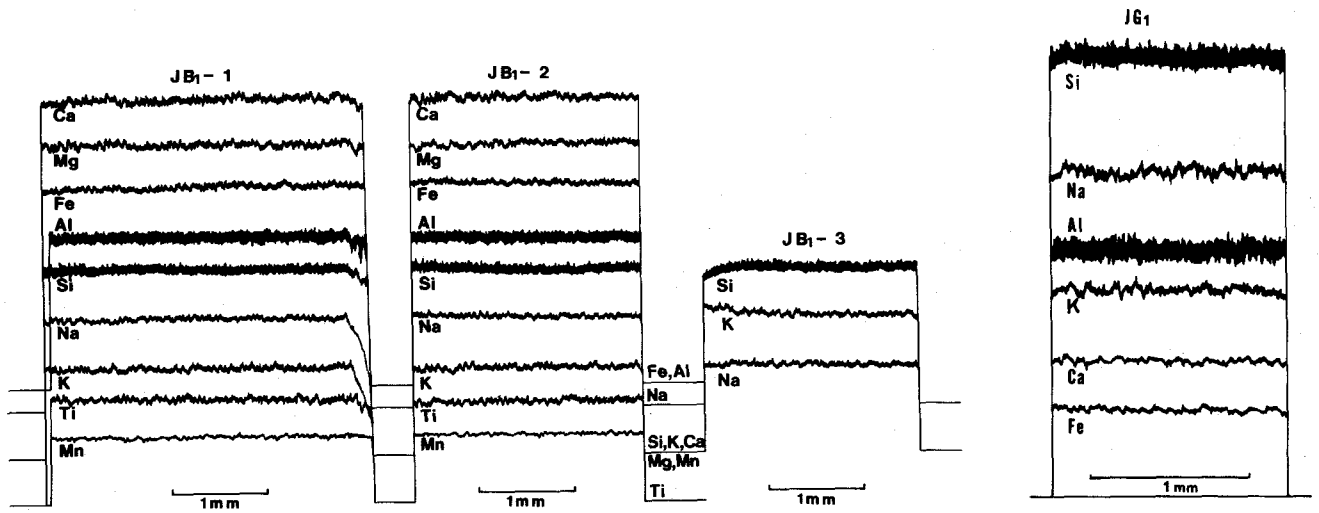

Fig. 1. Scanning profiles on the standard samples, JB–1 and JG–1.

specimen current on periclase and 40 μm electron beam diameter. A specimen was driven at 100 μm/min., during counting (100 seconds integration). Analyses of three to five parts were averaged for a glass analysis. The following crystals and glasses of known composition were used as standards; synthetic crystals: Al_2O_3 (Al), TiO_2 (Ti), MnO (Mn), MgO (Mg) and $CaSiO_3$ (Ca); synthetic glasses (Si, Fe, K); natural minerals: quartz (Si), hematite (Fe) and albite (Na). Correction procedures of microprobe data follow BENCE and ALBEE (1968), with the α factors of YAMAGUCHI *et al.* (1978).

III. Precision and accuracy

Standard rocks of Geological Survey of Japan, JB–1 and JG–1, were analysed by the procedure mentioned above. The total counts in 300 and 400 seconds for JB–1 and JG–1, respectively, and their specific errors of counting are shown in Table 1. The average, standard deviation and specific inaccuracy of 9 JB–1 and 5 JG–1 glasses are listed in Table 2. For most of the elements, the standard deviation is significantly larger than the count statistics. Compared with the recommended data of standard rocks (ANDO *et al.*, 1974), our data have specific inaccuracy less than 1% for most of elements. But for MgO and K_2O of JB–1, and TiO_2, MnO, MgO and Na_2O of JG–1, specific inaccuracy is rather large.

Table 1. Counts of measurement for the standard rock glasses and statistic errors.

	JB-1			JG-1		
	Intensity (cps)	Background (cps)	Specific* error of count (%)	Intensity (cps)	Background (cps)	Specific* error of count (%)
SI	3896.6	8.3	0.080	5577.7	8.7	0.077
TI	108.9	10.5	0.56	26.9	8.8	1.9
AL	1504.2	7.6	0.13	1518.5	6.9	0.15
FE	442.5	17.6	0.25	26.3	1.9	1.3
MN	26.8	19.1	4.4	2.2	1.3	12
MG	653.6	4.3	0.20	60.5	4.4	0.83
CA	742.7	5.7	0.19	143.1	5.3	0.51
NA	102.3	1.8	0.51	108.6	1.5	0.57
K	99.0	2.8	0.52	284.9	2.8	0.35

*: Specific error of count $= (Sp/t + Bg/t)^{1/2}/(Sp-Bg)$, where, Sp is total intensity (cps) of sample, Bg is background intensity (cps) and t is counting time interval.

Table 2. Analyses of JB-1 and JG-1 in comparison with their recommended mean (after ANDO *et al.*, 1974).

JB-1	Mean (N=9) X_1	σ	σ/X_1 (%)	Recommended mean X_2	Specific inaccuracy $(X_1-X_2)/X_1$ (%)
SiO_2	52.84	0.24	0.45	52.92	-0.15
TiO_2	1.36	0.015	1.1	1.37	-0.74
Al_2O_3	14.68	0.11	0.75	14.70	-0.14
FeO*	8.22	0.080	0.97	8.18	0.40
MnO	0.15	0.035	23	0.15	0
MgO	7.93	0.071	0.90	7.82	1.4
CaO	9.39	0.15	1.6	9.39	0
Na_2O	2.83	0.088	3.1	2.84	-0.35
K_2O	1.42	0.034	2.4	1.45	-2.1
JG-1	**Mean (N=5) X_1**	**σ**	**σ/X_1 (%)**	**Recommended mean X_2**	**Specific inaccuracy $(X_1-X_2)/X_1$ (%)**
SiO_2	73.15	0.26	0.36	72.71	0.60
TiO_2	0.29	0.017	5.9	0.26	9.7
Al_2O_3	14.19	0.099	0.70	14.32	-0.94
FeO*	2.00	0.033	1.7	1.98	0.80
MnO	0.076	0.008	11	0.062	18
MgO	0.79	0.037	4.7	0.74	6.1
CaO	2.22	0.063	2.8	2.21	0.63
Na_2O	3.32	0.14	4.2	3.4	-2.5
K_2O	3.99	0.045	1.1	3.98	0.20

*: Total Fe as FeO.

Fig. 2 shows comparison of the Na_2O and K_2O contents of 76 rock samples, determined by EPMA and flame photometry. These data obtained by the two independent methods show good agreement for each of alkalis. Vaporization of alkalis is, thus, concluded to be negligible.

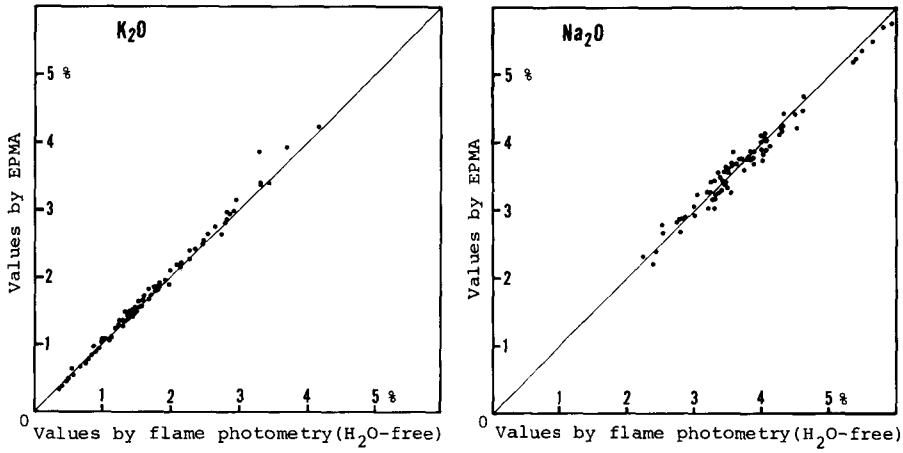

Fig. 2. Comparisons between the analyses by EPMA-method and those by flame photometry (H_2O-free).

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